Flow rate: 1.5
Detector: UV 225

CHROMATOGRAM
Retention time: 8.6

OTHER SUBSTANCES
Simultaneous: degradation products, liothyronine

REFERENCE
Won,C.M. Kinetics of degradation of levothyroxine in aqueous solution and in solid state, *Pharm.Res.*, **1992**, *9*, 131–137.

SAMPLE
Matrix: tissue
Sample preparation: 100 µL Thyroid tissue + 200 µL MeCN, mix, centrifuge. Remove a 100 µL aliquot of the supernatant and add it to 100 µL 4 nM dabsyl chloride in MeCN, heat at 70° for 10 min, add 400 µL MeOH:50 mM pH 7.0 phosphate buffer 50:50, inject a 20 µL aliquot.

HPLC VARIABLES
Column: 150 × 4.6 5 µm Hypersil ODS
Mobile phase: Gradient. A was MeOH:25 mM pH 6.5 sodium acetate 56:44. B was MeOH. A:B from 80:20 to 35:65 over 15 min, maintain at 35:65 for 3 min, to 0:100 over 1 min, maintain at 0:100 for 2 min.
Flow rate: 1
Injection volume: 20
Detector: UV 436

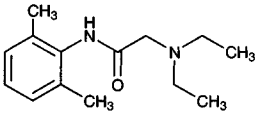
CHROMATOGRAM
Retention time: 17.5

OTHER SUBSTANCES
Extracted: diiodothyronine (T2), liothyronine (T3)

KEY WORDS
derivatization; thyroid

REFERENCE
Jansen,E.H.J.M.; van den Berg,R.H.; Both-Miedema,R.; Doorn,L. Advantages and limitations of pre-column derivatization of amino acids with dabsyl chloride, *J.Chromatogr.*, **1991**, *553*, 123–133.

Lidocaine



Molecular formula: $C_{14}H_{22}N_2O$
Molecular weight: 234.34
CAS Registry No.: 137-58-6, 6108-05-0 (HCl monohydrate), 73-78-9 (HCl)
Merck Index: 5505
Lednicer No.: 1 16

SAMPLE
Matrix: blood
Sample preparation: Mix 1 mL plasma with 200 µL 2 µg/mL IS in MeOH, add 2 mL water and 2 mL MeCN, vortex gently, set aside for 3 min, centrifuge at 2200 g for 20 min. Separate the clear supernatant, add 500 µL 200 mM NaOH and extract with 6 mL n-hexane by vortexing for 2 min. Centrifuge at 2200 g for 15 min. Evaporate 5 mL of the organic phase to dryness under reduced pressure. Reconstitute the residue in 120 µL mobile phase. Inject a 100 µL aliquot.

HPLC VARIABLES
Guard column: 10 × 3 5 μm AGP bonded silica (ChromTech, Hagersten, Sweden)
Column: 150 × 4 5 micro.m AGP bonded silica (ChromTech, Hagersten, Sweden)
Mobile phase: Isopropanol:buffer 4:96 (Prepare mobile phase by adding 4% isopropanol and 0.6% diethylamine to 8 mM sodium dihydrogen phosphate containing 100 mM NaCl, adjust to pH 7.05 with 50% phosphoric acid.)
Flow rate: 0.9
Injection volume: 100
Detector: UV 214

CHROMATOGRAM
Retention time: 7.5
Internal standard: diazepam (19.21)
Limit of detection: 10 ng/mL
Limit of quantitation: 12.5 ng/mL

OTHER SUBSTANCES
Extracted: bupivacaine

KEY WORDS
plasma; pharmacokinetics

REFERENCE
Abraham,I.; Fawcett,J.P.; Kennedy,J.; Kumar,A.; Ledger,R. Simultaneous analysis of lignocaine and bupivacaine enantiomers in plasma by high-performance liquid chromatography, *J.Chromatogr.B*, **1997**, *703*, 203–208.

SAMPLE
Matrix: blood
Sample preparation: Condition a 3 mL Bond-elut C2 SPE cartridge with 2 mL MeOH and 2 mL water. Apply 1 mL plasma to the cartridge, wash with 1 mL water, wash with 1 mL MeOH: water 50:50, wash with 1 mL MeCN, elute with 2 mL 1 M NaCl:MeOH 5:95. Dry the eluate under vacuum, resuspend in 200 μL MeOH, inject a 20 μL aliquot.

HPLC VARIABLES
Guard column: 10 mm long C18 (Waters)
Column: 250 × 4.6 Supelcosil LC-8-DB
Mobile phase: MeCN:20 mM phosphoric acid containing 200 μL/L triethylamine 10:90
Flow rate: 1.7
Detector: UV 263

CHROMATOGRAM
Retention time: 17.2
Internal standard: tocainide (9.7)
Limit of detection: 100 ng/mL
Limit of quantitation: 200 ng/L

OTHER SUBSTANCES
Extracted: metabolites
Noninterfering: acetaminophen, N-acetylprocainamide, amitriptyline, bupivicaine, caffeine, carbamazepine, chloramphenicol, cyclosporin A, desipramine, diazepam, disopyramide, doxepin, ethosuximide, flecainide, fluoxetine, ibuprofen, imipramine, naproxen, norchlordiazepoxide, nordiazepam, nordoxepine, nortriptyline, phenobarbital, phenytoin, primidone, procainamide, quinidine, salicylic acid, theophylline, valproic acid

KEY WORDS
serum; comparison with fluorescence polarization immunoassay; SPE

REFERENCE
O'Neal,C.L.; Poklis,A. Sensitive HPLC for simultaneous quantification of lidocaine and its metabolites monoethylglycinexylidide and glycinexylidide in serum, *Clin.Chem.*, **1996**, *42*, 330–331.

SAMPLE
Matrix: blood
Sample preparation: 1 mL Plasma + 50 μL 40 μg/mL etidocaine hydrochloride in water + 100 μL 1 M NaOH, vortex for 15 s, add 5 mL diethyl ether, shake on a reciprocating shaker for 10 min, centrifuge. Remove the organic layer and evaporate it to dryness under a stream of nitrogen, reconstitute the residue in 100 μL mobile phase, inject a 80 μL aliquot.

HPLC VARIABLES
Column: 150 × 3.2 10 μm μBondapak C18
Mobile phase: MeCN:50 mM pH 5.80 Na_2HPO_4 25:75
Flow rate: 0.9
Injection volume: 80
Detector: UV 210

CHROMATOGRAM
Retention time: 4.5
Internal standard: etidocaine (12.0)

OTHER SUBSTANCES
Extracted: 2,6-pipecolylxylidine, bupivacaine, mepivacaine
Noninterfering: metabolites, 2,3-chloroprocaine, theophylline, mexiletine, quinidine, disopyramide, verapamil, phenobarbital, phenytoin, carbamazepine, ethosuximide, digoxin, theobromine, caffeine, furosemide, phenprocoumon, aldactone

KEY WORDS
plasma

REFERENCE
Ha,H.-R.; Funk,B.; Gerber,H.R.; Follath,F. Determination of bupivacaine in plasma by high-performance liquid chromatography, *Anesth.Analg.*, **1984**, *63*, 448–450.

SAMPLE
Matrix: blood
Sample preparation: Filter plasma (0.22 μm), inject a 10 μL aliquot.

HPLC VARIABLES
Column: 150 × 4.6 5 μm GFF-S5-80 internal-surface reversed phase "Pinkerton" (Regis)
Mobile phase: THF:100 mM potassium phosphate 10:90, pH 7.2
Flow rate: 0.8
Injection volume: 10
Detector: UV 220

CHROMATOGRAM
Retention time: 12.9

KEY WORDS
plasma; direct injection

REFERENCE
Nakagawa,T.; Shibukawa,A.; Shimono,N.; Kawashima,T.; Tanaka,H.; Haginaka,J. Retention properties of internal-surface reversed-phase silica packing and recovery of drugs from human plasma, *J.Chromatogr.*, **1987**, *420*, 297–311.

SAMPLE
Matrix: blood
Sample preparation: 50 μL Serum + 100 μL 1 M pH 9.0 borate buffer + 1 mL chloroform:EtOH 82.5:17.5, mix, centrifuge. Remove the organic layer and evaporate it to dryness, reconstitute the residue in 50 μL mobile phase, inject an aliquot.

HPLC VARIABLES
Column: 300 × 2 10 μm μBondapak C18

Mobile phase: MeOH:MeCN:buffer 12:16:72 (Buffer was 31 mM sodium acetate adjusted to pH 5.1 with 40% phosphoric acid containing 0.15 mM tetrabutylammonium phosphate.)
Flow rate: 0.3
Injection volume: 20
Detector: UV 230

CHROMATOGRAM
Retention time: 10
Internal standard: lidocaine

OTHER SUBSTANCES
Extracted: caffeine, cocaine, metabolites
Simultaneous: barbital, phenobarbital, flumazepil, mazindol, hexobarbital, nicotine, procaine, cotinine
Noninterfering: amphetamine, desipramine, tetracaine, methadone, reserpine, buspirone, diazepam, haloperidol, chlordiazepoxide, oxazepam, midazolam, clonazepam, chlorpromazine, pentobarbital

KEY WORDS
serum; rat; lidocaine is IS

REFERENCE
Lau,C.E.; Ma,F.; Falk,J.L. Simultaneous determination of cocaine and its metabolites with caffeine in rat serum microsamples by high-performance liquid chromatography, *J.Chromatogr.*, **1990**, *532*, 95–103.

SAMPLE
Matrix: blood
Sample preparation: 1 mL Plasma + 100 μL 1 M NaOH + 3 mL heptane:ethyl acetate 90:10, shake for 2 min, centrifuge at 1200 g for 10 min. Remove the organic phase and add it to 50 μL 50 mM sulfuric acid, shake for 2 min, centrifuge at 1200 g for 5 min. Remove the aqueous phase and add it to 820 μg sodium acetate, inject a 40 μL aliquot. (The sodium acetate was measured out by adding 50 μL 200 mM sodium acetate in MeOH to the tube and evaporating the MeOH.)

HPLC VARIABLES
Column: 250 × 4 10 μm μBondapak C18
Mobile phase: MeCN:10 mM NaH_2PO_4 5:95, adjusted to pH 2.1
Column temperature: 30
Flow rate: 1
Injection volume: 40
Detector: UV 205

CHROMATOGRAM
Retention time: 13
Limit of detection: 5 ng/mL

OTHER SUBSTANCES
Extracted: procaine

KEY WORDS
plasma; rabbit

REFERENCE
Le Guévello,P.; Le Corre,P.; Chevanne,P.; Le Verge,R. High-performance liquid chromatographic determination of bupivacaine in plasma samples for biopharmaceutical studies and application to seven other local anaesthetics, *J.Chromatogr.*, **1993**, *622*, 284–290.

SAMPLE
Matrix: blood
Sample preparation: 1 mL Plasma + 100 μL 2 M NaOH, mix, add 3 mL n-hexane, shake for 1 min, centrifuge at 3500 rpm for 10 min. Remove the organic layer and evaporate it to dryness

with nitrogen under vacuum, reconstitute the residue in 200 μL mobile phase, vortex, inject a 100 μL aliquot.

HPLC VARIABLES
Column: 250 × 4 5 μm Spherisorb ODS-2
Mobile phase: MeOH:50 mM pH 5.9 KH_2PO_4 38:62
Flow rate: 1
Injection volume: 100
Detector: UV 254

CHROMATOGRAM
Retention time: 3.8
Internal standard: lidocaine

OTHER SUBSTANCES
Extracted: bupivacaine

KEY WORDS
plasma; lidocaine is IS

REFERENCE
Murillo,I.; Costa,J.; Salvá,P. Determination of bupivacaine in human plasma by HPLC, *J.Liq.Chromatogr.*, **1993**, *16*, 3509–3514.

SAMPLE
Matrix: blood
Sample preparation: Rock 5 mL whole blood + 10 mL water + 8.5 mL Na_2WO_4 in a 50 mL stoppered tube for 1 min, add 6 mL $NiCl_2$, rock for 5 min, add 15 mL dichloromethane:isobutyl alcohol:THF 30:45:25, centrifuge at 2500 g for 15 min. Remove organic phase and repeat the process. Filter all organic phases through a 40-90 μm filter and evaporate to dryness in a 100 mL porcelain dish at a moderate temperature in a sand bath. Take up residue in 500 μL MeCN: water 80:20, inject a 20 μL aliquot. (Na_2WO_4 prepared by mixing 10 g $Na_2WO_4.2H_2O$ in 38 mL of 2 M NaOH and 2.5 g of $NaHCO_3$ and making up to 100 mL. $NiCl_2$ was 17% w/v $NiCl_2$ in water.)

HPLC VARIABLES
Column: 200 × 4.6 5 μm Hypersil C8
Mobile phase: A = MeCN; B = 20 mM n-propylamine adjusted to pH 5 with 85% phosphoric acid. A:B from 15:85 to 20:80 over 5 min to 45:55 over another 15 min to 65:35 over another 5 min
Injection volume: 20
Detector: UV 230

CHROMATOGRAM
Retention time: 16
Limit of detection: 0.40 ppm

OTHER SUBSTANCES
Extracted: buprenorphine, caffeine, cocaine, codeine, diamorphine, ethylmorphine, methaqualone, morphine, naloxone, noscapine, papaverine, pentazocine, procaine
Also analyzed: bromazepam, clonazepam, diazepam, flunitrazepam, flurazepam, medazepam, nitrazepam, oxazepam

KEY WORDS
whole blood

REFERENCE
Bernal,J.L.; Del Nozal,M.J.; Rosas,V.; Villarino,A. Extraction of basic drugs from whole blood and determination by high performance liquid chromatography, *Chromatographia*, **1994**, *38*, 617–623.

SAMPLE
Matrix: blood
Sample preparation: 200 μL Plasma + 10 μL 15 mg/mL bupivacaine, mix, add 100 μL 2 M NaOH, vortex briefly, add 5 mL anhydrous ethyl ether, vortex for 30 s, rotate for 10 min, centrifuge at 1000 g for 5 min. Remove 4.5 mL ether and add to 250 μL 12.5 mM sulfuric acid, vortex for 30 s, rotate for 10 min, centrifuge for 5 min, inject a 50 μL aliquot of the lower aqueous phase.

HPLC VARIABLES
Column: 150 × 4.6 5 μm Octyl 1B (Keystone)
Mobile phase: MeCN:50 mM Na_2HPO_4 27:73 pH adjusted to 5.8 with 50% phosphoric acid
Flow rate: 1
Injection volume: 50
Detector: UV 210

CHROMATOGRAM
Retention time: 4.90
Internal standard: bupivacaine (9.81)
Limit of detection: 4 ng/mL
Limit of quantitation: 200 ng/mL

OTHER SUBSTANCES
Extracted: prilocaine, o-toluidine

KEY WORDS
plasma; pig; pharmacokinetics

REFERENCE
Klein,J.; Fernandes,D.; Gazarian,M.; Kent,G.; Koren,G. Simultaneous determination of lidocaine, prilocaine and the prilocaine metabolite o-toluidine in plasma by high-performance liquid chromatography, *J.Chromatogr.B*, **1994**, *655*, 83–88.

SAMPLE
Matrix: blood
Sample preparation: 100 μL Plasma + 100 μL 20 μg/mL caffeine + 8 mL dichloromethane, shake for 20 min, centrifuge at 2500 rpm for 20 min. Remove 7 mL of the organic layer and evaporate it to dryness under nitrogen or at 60°. Dissolve residue in 200 μL mobile phase, inject a 20 μL aliquot.

HPLC VARIABLES
Column: 150 × 6 Shimpack CLS-ODS (Shimadzu)
Mobile phase: MeCN:MeOH:0.5 mM phosphoric acid 7.5:3:89.5
Column temperature: 40
Flow rate: 1.5
Injection volume: 20
Detector: UV 210

CHROMATOGRAM
Internal standard: caffeine

KEY WORDS
plasma; rat

REFERENCE
Lee,C.K.; Uchida,T.; Kitagawa,K.; Yagi,A.; Kim,N.-S.; Goto,S. Skin permeability of various drugs with different lipophilicity, *J.Pharm.Sci.*, **1994**, *83*, 562–565.

SAMPLE
Matrix: blood

Sample preparation: Automated SPE by ASPEC system. Condition a C18 Clean-Up SPE cartridge (CEC 18111, Worldwide Monitoring) with 2 mL MeOH then 2 mL water. 1 mL Plasma + 1 mL 400 ng/mL protriptyline in water, vortex, add to column, wash with 3 mL water, wash with 3 mL 750 mL/L methanol. Elute with three aliquots of 300 μL 0.1 M ammonium acetate in MeOH. Add 0.5 mL 0.5 M NaOH and 4 mL 50 mL/L isopropanol in heptane to eluate, mix thoroughly. Allow 5 min for phase separation. Remove upper heptane phase and add it to 300 μL 0.1 M phosphoric acid (pH 2.5), mix, separate, inject a 100 μL aliquot of the aqueous phase.

HPLC VARIABLES

Guard column: LC-8-DB (Supelco)
Column: 150 × 4.6 LC-8-DB (Supelco)
Mobile phase: MeCN:buffer 35:65 (Buffer was 10 mL/L triethylamine in water adjusted to pH 5.5 with glacial acetic acid.)
Flow rate: 2
Injection volume: 100
Detector: UV 228

CHROMATOGRAM

Retention time: 1.8
Internal standard: protriptyline (4)

OTHER SUBSTANCES

Extracted: acetazolamide, amitriptyline, chlordiazepoxide, chlorimipramine, chlorpromazine, desipramine, dextromethorphan, diazepam, diphenhydramine, doxepin, fentanyl, flecainide, fluoxetine, flurazepam, haloperidol, hydroxyethylflurazepam, ibuprofen, imipramine, maprotiline, methadone, methaqualone, midazolam, norchlorimipramine, nordiazepam, nordoxepin, norfluoxetine, nortriptyline, norverapamil, promazine, propafenone, propoxyphene, protriptyline, temazepam, trazodone, trimipramine, verapamil
Noninterfering: acetaminophen, acetylmorphine, amiodarone, amobarbital, amphetamine, bendroflumethiazide, benzocaine, benzoylecgonine, benzthiazide, butalbital, carbamazepine, chlorothiazide, clonazepam, cocaine, codeine, cotinine, cyclosporine, cyclothiazide, desalkylflurazepam, diamorphine, dicumerol, ephedrine, ethacrynic acid, ethanol, ethchlorvynol, ethosuximide, furosemide, glutethimide, hydrochlorothiazide, hydrocodone, hydroflumethiazide, hydromorphone, lorazepam, mephentermine, meprobamate, methamphetamine, metharbital, methoxsalen, methoxyphenteramine, methsuximide, methylcyclothiazide, metoprolol, MHPG, monoacetylmorphine, morphine, normethsuximide, oxazepam, oxycodone, oxymorphone, pentobarbital, phencyclidine, phenteramine, phenylephrine, phenytoin, polythiazide, primidone, prochlorperazine, salicylic acid, sulfanilamide, THC-COOH, theophylline, thiazolam, thiopental, thioridazine, tocainide, trichloromethiazide, trifluoperazine, valproic acid, warfarin
Interfering: encainide, mexiletine, pentazocine, propranolol, quinidine

KEY WORDS

plasma; SPE

REFERENCE

Nichols,J.H.; Charlson,J.R.; Lawson,G.M. Automated HPLC assay of fluoxetine and norfluoxetine in serum, *Clin.Chem.*, **1994**, *40*, 1312–1316.

SAMPLE

Matrix: blood
Sample preparation: 1 mL Plasma + 100 μL 10 μg/mL bupivacaine in 25 mM sulfuric acid + 1 mL 1 M NaOH + 5 mL diethyl ether, shake or rotate for 15 min, centrifuge at 1000 rpm for 5 min, freeze at -20°. Remove the organic layer and add it to 250 μL 25 mM sulfuric acid, shake for 15 min, centrifuge at 1000 rpm for 5 min, freeze, discard the organic layer. Thaw the aqueous layer, pass air over the aqueous phase at room temperature to remove traces of ether, adjust pH to 5.0-6.5 by adding 10 μL 1 M NaOH, inject a 50 μL aliquot.

HPLC VARIABLES

Guard column: 4 × 4 4 μm LiChroCART Superspher 60 RP Select B (Merck)
Column: 125 × 4 4 μm LiChroCART Superspher 60 RP Select B (Merck)
Mobile phase: MeCN:buffer 30:70 (Buffer was 7.0 g/L K_2HPO_4 in water adjusted to pH 5.8 with 1 M NaOH.)

Column temperature: 40
Flow rate: 1
Injection volume: 50
Detector: UV 202

CHROMATOGRAM

Retention time: 5
Internal standard: bupivacaine (10)
Limit of quantitation: 100 ng/mL

KEY WORDS

plasma

REFERENCE

Sattler,A.; Krämer,I.; Jage,J.; Vrana,S.; Kleemann,P.P.; Dick,W. Development of a HPLC-system for quantitative measurement of lidocaine and bupivacaine in patients plasma during postoperative epidural pain therapy, *Pharmazie*, **1995**, *50*, 741–744.

SAMPLE

Matrix: blood
Sample preparation: 2 mL Whole blood or plasma + 2 mL buffer + 5 mL chloroform:isopropanol: n-heptane 60:14:26, shake gently horizontally for 10 min, centrifuge at 2800 g for 10 min. Remove the lower organic layer and evaporate it to dryness under vacuum at 45°, reconstitute the residue in 100 μL mobile phase, centrifuge at 2800 g for 5 min, inject a 50 μL aliquot of the supernatant. (Buffer was saturated ammonium chloride solution 25% diluted with water, adjusted to pH 9.5 with 25% ammonia solution.)

HPLC VARIABLES

Column: 300 × 3.9 4 μm NovaPack C18
Mobile phase: MeOH:THF:buffer 65:5:30 (Buffer was 0.68 g/L (10 mM (sic)) KH_2PO_4 adjusted to pH 2.6 with concentrated orthophosphoric acid.) (At the end of each session wash the column with water for 1 h and MeOH for 1 h, re-equilibrate for 30 min.)
Column temperature: 30
Flow rate: 0.8
Injection volume: 50
Detector: UV 264

CHROMATOGRAM

Retention time: 4.45
Limit of detection: <120 ng/mL

KEY WORDS

whole blood; plasma; interferences may occur–compounds (all of which are extracted) elute in this order tenoxicam; iproniazid; methocarbamol; methotrexate; caffeine; nialamide; colchicine; cytarabine; benzoylecgonine; acetaminophen; diazoxide; dacarbazine; sulfinpyrazole; flumazenil; sulpride; morphine; atenolol; toloxatone; terbutaline; albuterol; phenobarbital; ranitidine; tiapride; phenol; chlormezanone; aspirin; metformin; ritodrine; codeine; sultopride; amisulpride; naltrexone; lisinopril; benzocaine; nizatidine; nalorphine; mephenesin; naloxone; sotalol; carteolol; procainamide; carbamazepine; bromazepam; nalbuphine; nadolol; procarbazine; dihydralazine; omeprazole; strychnine; acebutolol; glutethimide; chlorpropamide; glipizide; triazolam; prazosin; flunitrazepam; clonazepam; metoclopramide; melphalan; estazolam; tolbutamide; ephedrine; clonidine; pindolol; clobazam; minoxidil; disopyramide; nitrazepam; dextromethorphan; tofisopam; zopiclone; debrisoquine; sulindac; alprazolam; cycloguanil; lorazepam; methaqualone; ketamine; piroxicam; metoprolol; nifedipine; quinine; mephentermine; prilocaine; pentazocine; oxazepam; tiaprofenic acid; quinidine; celiprolol; ajmaline; yohimbine; lidocaine; secobarbital; viloxazine; mepivacaine; meperidine; doxylamine; labetalol; temazepam; amodiaquine; benperidol; droperidol; hydroxychloroquine; zolpidem; ketoprofen; alminoprofen; cicletanine; moclobemide; chloroquine; cocaine; timolol; nomifensine; ticlopidine; acenocoumarol; vindesine; mexiletine; dipyridamole; trazodone; pipamperone; pyrimethamine; benazepril; vincristine; metapramine; chlordiazepoxide; oxprenolol; warfarin; clorazepate; flecainide; phencyclidine; thiopental; fenfluramine; metipranolol; triprolidine; naproxen; buprenorphine; verapamil; buspirone; tianeptine; midazolam; bupivacaine; carbinoxamine; loprazolam; cetirizine; chlorpheniramine; moperone; cibenzoline; medifoxamine; astemizole;

vinblastine; nicardipine; bisoprolol; diltiazem; glibornuride; reserpine; aconitine; nitrendipine; diazepam; mianserin; ramipril; haloperidol; tetracaine; alprenolol; acepromethazine; glibenclamide; chlorophenacinone; doxepin; nimodipine; diphenhydramine; cyclizine; histapyrrodine; phenylbutazone; demexiptiline; clozapine; proguanil; trifluperidol; medazepam; cyamemazine; bumadizone; suriclone; propranolol; acepromazine; dothiepin; dextromoramide; fenoprofen; dextropropoxyphene; loxapine; betaxolol; propafenone; promethazine; thioproperazine; methadone; amoxapine; quinupramine; opipramol; cyproheptadine; brompheniramine; mefenidramine; protriptyline; flurbiprofen; tetrazepam; zorubicin; prazepam; alimemazine; loperamide; imipramine; desipramine; levomepromazine; hydroxyzine; niflumic acid; penbutolol; fluvoxamine; pimozide; daunorubicin; indomethacin; maprotiline; tropatenine; etodolac; fluoxetine; amitriptyline; nortriptyline; tioclomarol; diclofenac; mefloquine; trimipramine; chlorambucil; lidoflazine; ibuprofen; floctafenine; alpidem; loratadine; chlorpromazine; clomipramine; carpipramine; thioridazine; fentiazac; clemastine; mefenamic acid; fluphenazine; prochlorperazine; penfluridol; bepridil; terfenadine; trifluoperazine

REFERENCE

Tracqui,A.; Kintz,P.; Mangin,P. Systematic toxicological analysis using HPLC/DAD, *J.Forensic Sci.*, **1995**, *40*, 254–262.

SAMPLE

Matrix: blood

Sample preparation: Condition a Bond Elut phenyl SPE cartridge with 4 mL MeOH and 4 mL water. Add 1 mL serum or 300-500 μL ultrafiltrate to the SPE cartridge, wash with 5 mL water, wash with 2 mL MeOH:water 5:95, wash with 2 mL EtOH:water 2.5:97.5, wash with 2 mL MeCN:water 10:90, elute with 1 mL MeCN:50 mM pH 2.4 phosphate buffer 25:75, inject a 100 μL aliquot of the eluate.

HPLC VARIABLES

Column: 300 × 4.6 μBondapak

Mobile phase: MeCN:50 mM pH 4.0 KH_2PO_4 25:75

Flow rate: 1.5

Injection volume: 100

Detector: UV 210

CHROMATOGRAM

Internal standard: lidocaine

OTHER SUBSTANCES

Extracted: bupivacaine

KEY WORDS

serum; ultrafiltrate; SPE; lidocaine is IS

REFERENCE

Mazoit,J.X.; Cao,L.S.; Samii,K. Binding of bupivacaine to human serum proteins, isolated albumin and isolated α-1-acid glycopotein. Differences between the two enantiomers are partly due to cooperativity, *J.Pharmacol.Exp.Ther.*, **1996**, *276*, 109–115.

SAMPLE

Matrix: blood, CSF, gastric contents, urine

Sample preparation: 200 μL Serum, urine, CSF, or gastric fluid + 300 μL reagent. Flush column A to waste with 500 μL 500 mM ammonium sulfate, inject sample onto column A, flush column A to waste with 500 μL 500 mM ammonium sulfate, backflush the contents of column A onto column B with mobile phase, monitor the effluent from column B. (Reagent was 8.05 M guanidine HCl and 1.02 M ammonium sulfate in water.)

HPLC VARIABLES

Column: A 40 μm preparative grade C18 (Analytichem); B 75 × 2.1 pellicular C18 (Whatman) + 250 × 4.6 5 μm C8 end-capped (Whatman)

Mobile phase: Gradient. A was 50 mM pH 4.5 KH_2PO_4. B was MeCN:isopropanol 80:20. A:B 90:10 for 1 min, to 30:70 over 20 min.

Column temperature: 50

Flow rate: 1.5
Detector: UV 220

CHROMATOGRAM
Retention time: 8.0
Internal standard: heptanophenone (19)

OTHER SUBSTANCES
Extracted: acetaminophen, allobarbital, azinphos, barbital, brallobarbitone, bromazepam, butethal, caffeine, carbamazepine, carbaryl, cephaloridine, chloramphenicol, chlordiazepoxide, chlorothiazide, chlorvinphos, clothiapine, cocaine, coomassie blue, desipramine, diazepam, diphenhydramine, dipipanone, ethylbromphos, flufenamic acid, formothion, griseofulvin, indomethacin, lorazepam, malathion, medazepam, midazolam, oxazepam, paraoxon, penicillin G, pentobarbital, prazepam, propoxyphene, prothiophos, quinine, salicylic acid, secobarbital, strychnine, sulfamethoxazole, theophylline, thiopental, thioridazine, trimethoprim

KEY WORDS
serum; column-switching

REFERENCE
Kruger,P.B.; Albrecht,C.F.De V.; Jaarsveld,P.P. Use of guanidine hydrochloride and ammonium sulfate in comprehensive in-line sorption enrichment of xenobiotics in biological fluids by high-performance liquid chromatography, *J.Chromatogr.*, **1993**, *612*, 191–198.

SAMPLE
Matrix: blood, tissue
Sample preparation: 10 g Whole blood or tissue + 10 mL 100 mM HCl + 100 μL 200 μg/mL procaine in ethyl acetate, homogenize, shake for 10 min, centrifuge at 6000 g for 10 min, add the supernatant to an Extrelut-20 SPE cartridge, let stand for 15 min, pass ammonia gas through the column, elute with 40 mL chloroform. Evaporate the eluate to dryness, reconstitute the residue in 1 mL mobile phase, dilute 10 times with mobile phase, inject a 50 μL aliquot. (Ammonia gas was generated by placing concentrated ammonia under reduced pressure and pulling the evolved ammonia through the column.)

HPLC VARIABLES
Column: 250 × 4.6 6 μm normal phase silica (BST)
Mobile phase: MeCN:100 mM pH 2 KH_2PO_4:water:THF:concentrated phosphoric acid 5.4:90:4.6:1:1
Flow rate: 2
Injection volume: 50
Detector: UV 230

CHROMATOGRAM
Retention time: 5.28
Internal standard: procaine (4.13)
Limit of detection: 250 ng/mL

OTHER SUBSTANCES
Simultaneous: benzocaine, bupivacaine, cocaine, tetracaine

KEY WORDS
whole blood; SPE; brain; liver

REFERENCE
Benkö,A.; Kimura,K. Toxicological analysis of lidocaine in biological materials by using HPLC, *Forensic Sci.Int.*, **1991**, *49*, 65–73.

SAMPLE
Matrix: blood, urine
Sample preparation: 2 mL Whole blood, plasma, or urine + 1 mL saturated sodium carbonate + 10 μL 100 μg/mL etidocaine, add to a 3 mL Extrelut SPE cartridge, elute with 15 mL di-

chloromethane. Evaporate eluate to dryness under a stream of nitrogen at 40°, reconstitute in 100 μL 10 mM HCl, add 3 mL diethyl ether, vortex for 20 s, centrifuge at 2800 g for 5 min, inject a 40 μL aliquot of the aqueous layer.

HPLC VARIABLES
Guard column: 5 × 6 μBondapak Guard Pak
Column: 300 × 3.9 10 μm μBondapak C18
Mobile phase: MeCN:100 mM ammonium acetate 50:50
Flow rate: 1.5
Injection volume: 40
Detector: UV 230

CHROMATOGRAM
Retention time: 8
Internal standard: etidocaine (14)
Limit of detection: 20 ng/mL

OTHER SUBSTANCES
Extracted: prilocaine, bupivacaine, dibucaine
Also analyzed: procaine, butacaine, tetracaine, p-aminobenzoic acid, articaine, o-toluidine, caffeine, amphetamine, ephedrine, epinephrine, morphine, monoacetylmorphine, diamorphine, ethylmorphine, codeine, acetylcodeine

KEY WORDS
whole blood; plasma; SPE

REFERENCE
Rop,P.P.; Grimaldi,F.; Bresson,M.; Fornaris,M.; Viala,A. Liquid chromatographic analysis of cocaine, benzoylecgonine, local anaesthetic agents and some of their metabolites in biological fluids, *J.Liq.Chromatogr.*, **1993**, *16*, 2797–2811.

SAMPLE
Matrix: blood, urine
Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 μL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) μL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

HPLC VARIABLES
Guard column: 20 mm long Symmetry C18
Column: 250 × 4.6 5 μm Symmetry C8 (Waters)
Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.
Column temperature: 30
Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)
Injection volume: 10-30
Detector: UV 200.5

CHROMATOGRAM
Retention time: 9.922

KEY WORDS
whole blood

REFERENCE
Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149–163.

SAMPLE
Matrix: bulk
Sample preparation: Prepare a 10 mg/mL solution in 500 mM sodium bicarbonate solutions, extract a 10 mL aliquot twice with 15 mL portions of dichloromethane. Combine the extracts and add 10 μL phenylisothiocyanate, evaporate to dryness under a stream of air, reconstitute with 10 mL MeOH, inject a 10 μL aliquot.

HPLC VARIABLES
Guard column: 70 × 2.1 CO:PELL ODS
Column: 300 × 3.9 μBondapak C18
Mobile phase: MeOH:water:acetic acid 45:54:1
Flow rate: 2
Injection volume: 10
Detector: UV 254

CHROMATOGRAM
Retention time: 2

OTHER SUBSTANCES
Simultaneous: ephedrine, phenylpropanolamine, pseudoephedrine

KEY WORDS
derivatization

REFERENCE
Noggle,F.T.,Jr.; Clark,C.R. Liquid chromatographic analysis of samples containing cocaine, local anesthetics, and other amines, *J.Assoc.Off.Anal.Chem.*, **1983**, *66*, 151–157.

SAMPLE
Matrix: bulk
Sample preparation: Prepare a 750 μg/mL solution in 10 mM pH 2.5 orthophosphoric acid, sonicate for 10 min, filter (0.2 μm), inject a 15 μL aliquot.

HPLC VARIABLES
Guard column: 4 × 4 5 μm LiChrospher 100
Column: 125 × 4 3 μm Spherisorb ODS-1
Mobile phase: Gradient. A was water containing 5 mL/L 85% orthophosphoric acid and 0.56 mL/L hexylamine. B was MeCN:water 90:10 containing 5 mL/L 85% orthophosphoric acid and 0.56 mL/L hexylamine. A:B from 91:9 to 86:14 over 4 min, maintain at 86:14 for 13 min, to 55:45 over 11 min, maintain at 55:45 for 8 min, re-equilibrate at initial conditions for 20 min.
Flow rate: 0.7
Injection volume: 15
Detector: UV 210

CHROMATOGRAM
Retention time: 9.4

OTHER SUBSTANCES
Simultaneous: acetaminophen, acetylcodeine, benzocaine, caffeine, cocaine, codeine, diamorphine, 6-monoacetylmorphine, morphine, noscapine, papaverine, procaine

REFERENCE
Grogg-Sulser,K.; Helmlin,H.-J.; Clerc,J.-T. Qualitative and quantitative determination of illicit heroin street samples by reversed-phase high-performance liquid chromatography: method development by CARTAGO-S, *J.Chromatogr.A*, **1995**, *692*, 121–129.

SAMPLE
Matrix: formulations
Sample preparation: Dilute with MeOH:water 500 mM pH 7 sodium borate 35:65:2, inject an aliquot.

HPLC VARIABLES
Column: 250 × 4.6 10 μm Whatman PXS ODS-3 C18
Mobile phase: MeCN:buffer 20:80 (Buffer was water glacial acetic acid 93:5 adjusted to pH 3.0 with 1 M NaOH.)
Flow rate: 1.5
Injection volume: 20
Detector: UV 254

CHROMATOGRAM
Retention time: 5

OTHER SUBSTANCES
Simultaneous: milrinone

KEY WORDS
injections; 10% calcium chloride; 7.5% sodium bicarbonate; stability-indicating

REFERENCE
Wilson,T.D.; Forde,M.D. Stability of milrinone and epinephrine, atropine sulfate, lidocaine hydrochloride, or morphine sulfate injection, *Am.J.Hosp.Pharm.*, **1990**, *47*, 2504–2507.

SAMPLE
Matrix: perfusate

HPLC VARIABLES
Column: 100 × 8 4 μm Novapak C18
Mobile phase: MeCN:0.092%phosphoric acid + 0.2% triethylamine 26:74
Flow rate: 2
Detector: UV 214

CHROMATOGRAM
Internal standard: lidocaine
Limit of quantitation: 10 ng/mL

OTHER SUBSTANCES
Simultaneous: diphenhydramine, diltiazem, metabolites
Also analyzed: bupivacaine

KEY WORDS
lidocaine is IS; rat; liver

REFERENCE
Hussain,M.D.; Tam,Y.K.; Gray,M.R.; Coutts,K.T. Kinetic interactions of lidocaine, diphenhydramine, and verapamil with diltiazem: A study using isolated perfused rat liver, *Drug Metab.Dispos.*, **1994**, *22*, 530–536.

SAMPLE
Matrix: perfusate

HPLC VARIABLES
Column: 125 × 4 5 μm LiChrospher 60 RP-Select B C18
Mobile phase: Gradient. MeCN:buffer 9:91 for 5 min, to 19:81 over 10 min, maintain at 19:81 for 11.5 min, return to initial conditions over 1 min, re-equilibrate for 7.5 min. (Buffer was 6.66 g/L KH_2PO_4 , 150 μL/L phosphoric acid, and 5 mM sodium n-heptanesulfonate, pH 3.5.)
Flow rate: 1

Detector: UV 214

CHROMATOGRAM
Limit of quantitation: 25 ng/mL

KEY WORDS
rat; liver

REFERENCE
Ngo,L.Y.; Tam,Y.K.; Coutts,R.T. Lack of residual effects of diethyl ether, methoxyflurane, and sodium pentobarbital on lidocaine metabolism in a single-pass isolated rat liver perfusion system, *Drug Metab.Dispos.*, **1995**, *23*, 525–528.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 250 × 4 ODS (Hitachi)
Mobile phase: MeCN:50 mM phosphoric acid 40:60 containing 100 mM KCl
Column temperature: 55
Flow rate: 0.6
Injection volume: 20
Detector: UV 262

OTHER SUBSTANCES
Also analyzed: disopyramide, metoprolol

REFERENCE
Sugawara,M.; Takekuma,Y; Yamada,H.; Kobayashi,M.; Iseki,K.; Miyazaki,K. A general approach for the prediction of the intestinal absorption of drugs: regression analysis using the physicochemical properties and drug-membrane electrostatic interactions, *J.Pharm.Sci.*, **1998**, *87*, 960–966.

SAMPLE
Matrix: solutions
Sample preparation: Inject a 5 μL aliquot.

HPLC VARIABLES
Column: 300 × 4 10 μm μBondapak C18
Mobile phase: MeCN:MeOH:water 20:20:60 containing 0.06% sulfuric acid, 0.5% sodium sulfate, and 0.02% sodium heptanesulfonate, pH 2.6
Flow rate: 2
Injection volume: 5
Detector: UV 305

CHROMATOGRAM
Retention time: 2

OTHER SUBSTANCES
Simultaneous: benzocaine, butamben, pramoxine, procaine, tetracaine

REFERENCE
Menon,G.N.; Norris,B.J. Simultaneous determination of tetracaine and its degradation product, p-n-butylaminobenzoic acid, by high-performance liquid chromatography, *J.Pharm.Sci.*, **1981**, *70*, 569–570.

SAMPLE
Matrix: solutions
Sample preparation: Prepare a 10 μg/mL solution in MeOH, inject a 20 μL aliquot.

HPLC VARIABLES
Column: 125 × 4.9 Spherisorb S5W silica

Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7
Flow rate: 2
Injection volume: 20
Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM
Retention time: 1.4

OTHER SUBSTANCES
Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipanone, diprenorphine, dipyridamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserin, laudanosine, lofepramine, loxapine, maprotiline, mecamylamine, meclophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, piminodine, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranylcypromine, trazodone, trifluoperazine, trifluperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripelennamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE
Jane,I.; McKinnon,A.; Flanagan,R.J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J.Chromatogr.*, **1985**, *323*, 191–225.

SAMPLE
Matrix: solutions
Sample preparation: Dissolve in MeOH:water 1:1 at a concentration of 50 μg/mL, inject a 10 μL aliquot.

HPLC VARIABLES
Column: 300 × 3.9 10 μm μBondapak C18
Mobile phase: MeOH:acetic acid:triethylamine:water 50:1.5:0.5:48
Flow rate: 1.5
Injection volume: 10
Detector: UV 254

CHROMATOGRAM
Retention time: 6

OTHER SUBSTANCES
Simultaneous: butacaine, bupivacaine, benzocaine, tetracaine

REFERENCE
Roos,R.W.; Lau-Cam,C.A. General reversed-phase high-performance liquid chromatographic method for the separation of drugs using triethylamine as a competing base, *J.Chromatogr.*, **1986**, *370*, 403–418.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 250 × 4.6 Nucleosil 5C18
Mobile phase: MeCN:buffer 35:65 (Buffer was 0.1% phosphoric acid containing 5 mM sodium 1-hexanesulfonate.)
Flow rate: 1
Detector: UV 230

CHROMATOGRAM
Internal standard: ethyl p-hydroxybenzoate

REFERENCE
Cheng,Y.H.; Hosoya,O.; Sugibayashi,K.; Morimoto,Y. Effect of skin surface lipid on the skin permeation of lidocaine from pressure sensitive adhesives, *Biol.Pharm.Bull.*, **1994**, *17*, 1640–1644.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 250 × 4.6 Zorbax RX
Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.
Column temperature: 30
Flow rate: 2
Detector: UV 210

OTHER SUBSTANCES
Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitriptyline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepan, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlordiazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fencamfamine, fenoprofen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiacol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, iminostilbene, imipramine, indomethacin, isocarbostyril, isocarboxazid, isoniazid, isoproterenol, isoxsuprine, ivermectin, ketamine, ketoprofen, kynurenic acid, lorazepam, lormetazepam, lox-

apine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephenytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, methapyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyldopa, methyldopamine, methylphenidate, methylprednisolone, methyltestosterone, methyprylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, oxymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phenazopyridine, phencyclidine, phendimetrazine, phenelzine, pheniramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrilamine, pyrithyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfamerazine, sulfamethazine, sulfamethoxizole, sulfanilamide, sulfapyridine, sulfasoxizole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranylcypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripelennamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE

Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233–242.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 × 4.6 5 μm Supelcosil LC-DP (A) or 250 × 4 5 μm LiChrospher 100 RP-8 (B)
Mobile phase: MeCN:0.025% phosphoric acid:buffer 25:10:5 (A) or 60:25:15 (B) (Buffer was 9 mL concentrated phosphoric acid and 10 mL triethylamine in 900 mL water, adjust pH to 3.4 with dilute phosphoric acid, make up to 1 L.)
Flow rate: 0.6
Injection volume: 25
Detector: UV 229

CHROMATOGRAM

Retention time: 8.04 (A), 4.50 (B)

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetaminophen, acetazolamide, acetophenazine, albuterol, alprazolam, amitriptyline, amobarbital, amoxapine, antipyrine, atenolol, atropine, azatadine, baclofen, benzocaine, bromocriptine, brompheniramine, brotizolam, bupivacaine, buspirone, butabarbital, butalbital, caffeine, carbamazepine, cetirizine, chlorcyclizine, chlordiazepoxide, chlormezanone, chloroquine, chlorpheniramine, chlorpromazine, chlorpropamide, chlorprothixene, chlorthalidone, chlorzoxazone, cimetidine, cisapride, clomipramine, clonazepam, clonidine, clozapine, cocaine, codeine, colchicine, cyclizine, cyclobenzaprine, dantrolene, desipramine, diazepam, diclofenac, diflunisal, diltiazem, diphenhydramine, diphenidol, diphenoxylate, dipyridamole, disopyramide, dobutamine, doxapram, doxepin, droperidol, encainide, ethidium bromide, ethopropazine, fenoprofen, fentanyl, flavoxate, fluoxetine, fluphenazine, flurazepam, flurbiprofen, fluvoxamine, furosemide, glutethimide, glyburide, guaifenesin, haloperidol, homatropine, hydralazine, hydrochlorothiazide, hydrocodone, hydromorphone, hydroxychloroquine, hydroxyzine, ibuprofen, imipramine, indomethacin, ketoconazole, ketoprofen, ketorolac, labetalol, levorphanol, loratadine, lorazepam, lovastatin, loxapine, mazindol, mefenamic acid, meperidine, mephenytoin, mepivacaine, mesoridazine, metaproterenol, methadone, methdilazine, methocarbamol, methotrexate, methotrimeprazine, methoxamine, methyldopa, methylphenidate, metoclopramide, metolazone, metoprolol, metronidazole, midazolam, moclobemide, morphine, nadolol, nalbuphine, naloxone, naphazoline, naproxen, nifedipine, nizatidine, norepinephrine, nortriptyline, oxazepam, oxycodone, oxymetazoline, paroxetine, pemo-

line, pentazocine, pentobarbital, pentoxifylline, perphenazine, pheniramine, phenobarbital, phenol, phenolphthalein, phentolamine, phenylbutazone, phenyltoloxamine, phenytoin, pimozide, pindolol, piroxicam, pramoxine, prazepam, prazosin, probenecid, procainamide, procaine, prochlorperazine, procyclidine, promazine, promethazine, propafenone, propantheline, propiomazine, propofol, propranolol, protriptyline, quazepam, quinidine, quinine, racemethorphan, ranitidine, remoxipride, risperidone, salicylic acid, scopolamine, secobarbital, sertraline, sotalol, spironolactone, sulfinpyrazone, sulindac, temazepam, terbutaline, terfenadine, tetracaine, theophylline, thiethylperazine, thiopental, thioridazine, thiothixene, timolol, tocainide, tolbutamide, tolmetin, trazodone, triamterene, triazolam, trifluoperazine, triflupromazine, trimeprazine, trimethoprim, trimipramine, verapamil, warfarin, xylometazoline, yohimbine, zopiclone

KEY WORDS
details of plasma extraction

REFERENCE
Koves,E.M. Use of high-performance liquid chromatography-diode array detection in forensic toxicology, *J.Chromatogr.A*, **1995**, *692*, 103–119.

SAMPLE
Matrix: solutions
Sample preparation: Inject a 20 μL aliquot of a 100-500 μg/mL solution in mobile phase.

HPLC VARIABLES
Column: 100 × 4.6 5 μm Hypersil C8 MOS 100A coated with phosphatidylcholine (95% pure soybean lecithin, Epikuron, Lucas Meyer & Co.) (Coat column by recycling a 1 mM solution of phosphatidylcholine in MeOH:water 80:20 for 24 h.)
Mobile phase: MeCN:35 mM pH 7.4 sodium phosphate buffer 40:60
Flow rate: 0.5–2
Injection volume: 20
Detector: UV 254

CHROMATOGRAM
Retention time: k' 5.37

OTHER SUBSTANCES
Also analyzed: amoxicillin, antipyrine, carbamazepine, chlorpheniramine, chlorpromazine, clonidine, codeine, desipramine, diphenhydramine, dipyridamole, ephedrine, flufenamic acid, haloperidol, hydroxyzine, imipramine, indomethacin, megestrol acetate, metoprolol, nabumetone, nadolol, phenobarbital, phenol, promazine, propranolol, pyrilamine, quinidine, ropinirole, testosterone, thioridazine, tolfenamic acid, verapamil
Noninterfering: acetaminophen, aspirin, azathioprine, caffeine, carprofen, chlorambucil, cimetidine, fenoterol, flurbiprofen, ibuprofen, ketoprofen, ranitidine, salicylic acid, sulfamethoxazole, theophylline, thioguanine, tiaprofenic acid, trimethoprim, valproic acid

KEY WORDS
comparison with capillary electrophoresis

REFERENCE
Hanna,M.; de Biasi,V.; Bond,B.; Salter,C.; Hutt,A.J.; Camilleri,P. Estimation of the partitioning characteristics of drugs: A comparison of a large and diverse drug series utilizing chromatographic and electrophoretic methodology, *Anal.Chem.*, **1998**, *70*, 2092–2099.

SAMPLE
Matrix: urine
Sample preparation: 500 μL Urine + N-ethylnordiazepam + chlorpheniramine + 100 μL buffer, centrifuge at 11000 g for 30 s, inject a 500 μL aliquot onto column A with mobile phase A, after 0.6 min backflush column A with mobile phase A to waste for 1.6 min, elute column A with 250 μL mobile phase B, with 200 μL mobile phase C, and with 1.15 mL mobile phase D. Elute column A to waste until drugs start to emerge then elute onto column B. Elute column B to waste until drugs started to emerge, then elute onto column C. When all the drugs have emerged from column B remove it from the circuit, elute column C with mobile phase D,

monitor the effluent from column C. Flush column A with 7 mL mobile phase E, with mobile phase D, and mobile phase A. Flush column B with 5 mL mobile phase E then with mobile phase D. (Buffer was 6 M ammonium acetate adjusted to pH 8.0 with 2 M KOH.)

HPLC VARIABLES

Column: A 10 × 2.1 12-20 μm PRP-1 spherical poly(styrene-divinylbenzene) (Hamilton); B 10 × 3.2 11 μm Aminex A-28 (Bio-Rad); C 25 × 3.2 5 μm C8 (Phenomenex) + 150 × 4.6 5 μm silica (Macherey-Nagel)
Mobile phase: A 0.1% pH 8.0 potassium borate buffer; B 6 mM KH_2PO_4 containing 5 mM tetramethylammonium hydroxide, and 2 mM dimethyloctylamine, pH adjusted to 6.50 with phosphoric acid; C MeCN:buffer 40:60 (Buffer was 6 mM KH_2PO_4 containing 5 mM tetramethylammonium hydroxide, and 2 mM dimethyloctylamine, pH adjusted to 6.50 with phosphoric acid.); D MeCN:buffer 33:67 (Buffer was 6 mM KH_2PO_4 containing 5 mM tetramethylammonium hydroxide, and 2 mM dimethyloctylamine, pH adjusted to 6.50 with phosphoric acid.); E MeCN:buffer 70:30 (Buffer was 6 mM KH_2PO_4 containing 5 mM tetramethylammonium hydroxide, and 2 mM dimethyloctylamine, pH adjusted to 6.50 with phosphoric acid.)
Column temperature: ambient (column A), 40 (columns B and C)
Flow rate: A 5; B-E 1
Injection volume: 500
Detector: UV 210, UV 235

CHROMATOGRAM

Retention time: k' 2.7
Internal standard: N-ethylnordiazepam (k' 2.1), chlorpheniramine (k' 5.9)
Limit of detection: 300 ng/mL

OTHER SUBSTANCES

Extracted: methamphetamine, desipramine, nortriptyline, diphenhydramine, methadone, imipramine, flurazepam, amitriptyline, morphine, codeine, hydromorphone, hydrocodone, caffeine, cotinine, benzoylecgonine, secobarbital, oxazepam, phenobarbital, nordiazepam, diazepam, phenylpropanolamine
Interfering: phentermine, amphetamine, phenmetrazine, ephedrine, pentazocine

KEY WORDS

column-switching

REFERENCE

Binder,S.R.; Regalia,M.; Biaggi-McEachern,M.; Mazhar,M. Automated liquid chromatographic analysis of drugs in urine by on-line sample cleanup and isocratic multi-column separation, *J.Chromatogr.*, **1989**, *473*, 325–341.

SAMPLE

Matrix: urine
Sample preparation: 1 mL Urine + 0.5 mL 1% trichloroacetic acid, centrifuge at 5200 g for 10 min, filter (0.2 μm), inject 20 μL aliquot

HPLC VARIABLES

Column: 250 × 4 Lichrospher 5μm 60 RP-select B
Mobile phase: Gradient. MeCN:50 mM pH 3.2 potassium phosphate buffer from 10:90 to 50:50 over 15 min.
Flow rate: 1.5
Injection volume: 20
Detector: UV 190-370

CHROMATOGRAM

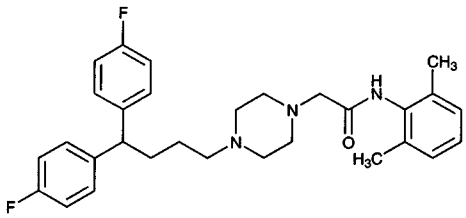
Retention time: 8

OTHER SUBSTANCES

Extracted: morphine, ephedrine, phenylpropanolamine, diphenhydramine, nortriptyline, cocaine, benzoylecgonine, norpropoxyphene, nordiazepam
Also analyzed: amitriptyline, amphetamine, meperidine, codeine, (different gradient)

REFERENCE
Li,S.; Gemperline,P.J.; Briley,K.; Kazmierczak,S. Identification and quantitation of drugs of abuse in urine using the generalized rank annihilation method of curve resolution, *J.Chromatogr.B*, **1994**, *655*, 213–223.

Lidoflazine



Molecular formula: $C_{30}H_{35}F_2N_3O$
Molecular weight: 491.62
CAS Registry No.: 3416-26-0
Merck Index: 5507
Lednicer No.: 1 279

SAMPLE
Matrix: blood
Sample preparation: 2 mL Whole blood or plasma + 2 mL buffer + 5 mL chloroform:isopropanol: n-heptane 60:14:26, shake gently horizontally for 10 min, centrifuge at 2800 g for 10 min. Remove the lower organic layer and evaporate it to dryness under vacuum at 45°, reconstitute the residue in 100 µL mobile phase, centrifuge at 2800 g for 5 min, inject a 50 µL aliquot of the supernatant. (Buffer was saturated ammonium chloride solution 25% diluted with water, adjusted to pH 9.5 with 25% ammonia solution.)

HPLC VARIABLES
Column: 300 × 3.9 4 µm NovaPack C18
Mobile phase: MeOH:THF:buffer 65:5:30 (Buffer was 0.68 g/L (10 mM (sic)) KH_2PO_4 adjusted to pH 2.6 with concentrated orthophosphoric acid.) (At the end of each session wash the column with water for 1 h and MeOH for 1 h, re-equilibrate for 30 min.)
Column temperature: 30
Flow rate: 0.8
Injection volume: 50
Detector: UV 265

CHROMATOGRAM
Retention time: 10.35
Limit of detection: <120 ng/mL

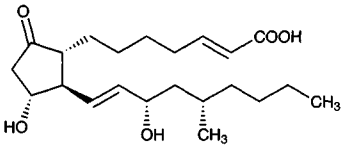
KEY WORDS
whole blood; plasma; interferences may occur–compounds (all of which are extracted) elute in this order tenoxicam; iproniazid; methocarbamol; methotrexate; caffeine; nialamide; colchicine; cytarabine; benzoylecgonine; acetaminophen; diazoxide; dacarbazine; sulfinpyrazole; flumazenil; sulpride; morphine; atenolol; toloxatone; terbutaline; albuterol; phenobarbital; ranitidine; tiapride; phenol; chlormezanone; aspirin; metformin; ritodrine; codeine; sultopride; amisulpride; naltrexone; lisinopril; benzocaine; nizatidine; nalorphine; mephenesin; naloxone; sotalol; carteolol; procainamide; carbamazepine; bromazepam; nalbuphine; nadolol; procarbazine; dihydralazine; omeprazole; strychnine; acebutolol; glutethimide; chlorpropamide; glipizide; triazolam; prazosin; flunitrazepam; clonazepam; metoclopramide; melphalan; estazolam; tolbutamide; ephedrine; clonidine; pindolol; clobazam; minoxidil; disopyramide; nitrazepam; dextromethorphan; tofisopam; zopiclone; debrisoquine; sulindac; alprazolam; cycloguanil; lorazepam; methaqualone; ketamine; piroxicam; metoprolol; nifedipine; quinine; mephentermine; prilocaine; pentazocine; oxazepam; tiaprofenic acid; quinidine; celiprolol; ajmaline; yohimbine; lidocaine; secobarbital; viloxazine; mepivacaine; meperidine; doxylamine; labetalol; temazepam; amodiaquine; benperidol; droperidol; hydroxychloroquine; zolpidem; ketoprofen; alminoprofen; cicletanine; moclobemide; chloroquine; cocaine; timolol; nomifensine; ticlopidine; acenocoumarol; vindesine; mexiletine; dipyridamole; trazodone; pipamperone; pyrimethamine; benazepril; vincristine; metapramine; chlordiazepoxide; oxprenolol; warfarin; clorazepate; flecainide; phencyclidine; thiopental; fenfluramine; metipranolol; triprolidine; naproxen; buprenorphine; verapamil; buspirone; tianeptine; midazolam; bupivacaine; carbinoxamine; loprazolam; cetirizine; chlorpheniramine; moperone; cibenzoline; medifoxamine; astemizole; vinblastine; nicardipine; bisoprolol; diltiazem; glibornuride; reserpine; aconitine; nitrendipine; diazepam; mianserin; ramipril; haloperidol; tetracaine; alprenolol; aceprometazine; glibencla-

mide; chlorophenacinone; doxepin; nimodipine; diphenhydramine; cyclizine; histapyrrodine; phenylbutazone; demexiptiline; clozapine; proguanil; trifluperidol; medazepam; cyamemazine; bumadizone; suriclone; propranolol; acepromazine; dothiepin; dextromoramide; fenoprofen; dextropropoxyphene; loxapine; betaxolol; propafenone; promethazine; thioproperazine; methadone; amoxapine; quinupramine; opipramol; cyproheptadine; brompheniramine; mefenidramine; protriptyline; flurbiprofen; tetrazepam; zorubicin; prazepam; alimemazine; loperamide; imipramine; desipramine; levomepromazine; hydroxyzine; niflumic acid; penbutolol; fluvoxamine; pimozide; daunorubicin; indomethacin; maprotiline; tropatenine; etodolac; fluoxetine; amitriptyline; nortriptyline; tioclomarol; diclofenac; mefloquine; trimipramine; chlorambucil; lidoflazine; ibuprofen; floctafenine; alpidem; loratadine; chlorpromazine; clomipramine; carpipramine; thioridazine; fentiazac; clemastine; mefenamic acid; fluphenazine; prochlorperazine; penfluridol; bepridil; terfenadine; trifluoperazine

REFERENCE

Tracqui,A.; Kintz,P.; Mangin,P. Systematic toxicological analysis using HPLC/DAD, *J.Forensic Sci.*, **1995**, *40*, 254–262.

Limaprost



Molecular formula: $C_{22}H_{36}O_5$
Molecular weight: 380.52
CAS Registry No.: 74397-12-9, 88852-12-4
Merck Index: 5514

SAMPLE

Matrix: blood

Sample preparation: Condition a Bond Elut Certify C18 SPE cartridge with water, MeCN, and 20 mM citric acid. Add 1 mL plasma to SPE cartridge, wash with 1 mL 20 mM citric acid, wash with 2 mL MeOH:water 10:90, wash with 2 mL cyclohexane, elute with 3 mL 3% ammonia in MeOH. Evaporate the eluate to dryness under a stream of nitrogen, reconstitute the residue in 500 μL MeCN, add 200 μL 10 mM DBD-PZ in MeCN, add 300 μL 10 mM 2,2'-dipyridyl disulfide and 10 mM triphenylphosphine in MeCN, let stand at room temperature for 30 min, inject an aliquot. (DBD-PZ prepared from 123 mg 4-(N,N-dimethylaminosulfonyl)-7-fluoro-2,1,3-benzoxadiazole in 20 mL MeCN added dropwise to 129 mg piperazine in 20 mL MeCN at room temperature, stir for 30 min, evaporate under reduced pressure, dissolve residue in 50 mL 5% HCl, extract three times with 20 mL ethyl acetate, discard ethyl acetate extracts, adjust pH of aqueous solution to 13-14 with 5% NaOH, extract five times with 50 mL ethyl acetate, combine extracts, wash with 20 mL water, dry over anhydrous sodium sulfate, evaporate under vacuum to give 4-(N,N-dimethylaminosulfonyl)-7-(1-piperazinyl)-2,1,3-benzoxadiazole (DBD-PZ) as orange crystals, mp 121-2° (J. Chromatogr. 1991, 588, 61).)

HPLC VARIABLES

Column: 150 × 4.6 5 μm Inertsil ODS-2
Mobile phase: Gradient. MeCN:water from 35:65 to 60:40 over 1 h
Column temperature: 40
Flow rate: 1
Detector: F ex 440 em 569

CHROMATOGRAM

Retention time: 37.5
Limit of detection: 1.7-5 fmole

OTHER SUBSTANCES

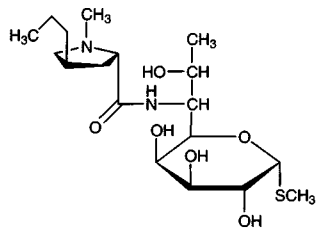
Extracted: alprostadil (prostaglandin E1), dinoprost (prostaglandin F2α), dinoprostone (prostaglandin E2), 6-ketoprostaglandin F1α, prostaglandin F1α, prostaglandin D2, prostaglandin A1, prostaglandin B1

KEY WORDS

plasma; rat; SPE; derivatization

REFERENCE
Toyo'oka,T.; Ishibashi,M.; Terao,T.; Imai,K. Sensitive fluorometric detection of prostaglandins by high performance liquid chromatography after precolumn labelling with 4-(*N,N*-dimethylaminosulphonyl)-7-(1-piperazinyl)-2,1,3-benzoxadiazole (DBD-PZ), *Biomed.Chromatogr.*, **1992**, *6*, 143–148.

Lincomycin



Molecular formula: $C_{18}H_{34}N_2O_6S$
Molecular weight: 406.54
CAS Registry No.: 154-21-2, 7179-49-9 (HCl monohydrate), 859-18-7 (HCl)
Merck Index: 5525

SAMPLE
Matrix: solutions
Sample preparation: Centrifuge and filter cell solutions (0.22 μm), inject an aliquot.

HPLC VARIABLES
Guard column: Guard-PAK C18 (Waters)
Column: 150 × 3.9 5 μm NOVA PAK C18
Mobile phase: MeCN:50 mM pH 6.0 KH_2PO_4 12:88
Flow rate: 1.5
Detector: UV 214

CHROMATOGRAM
Retention time: 3.9

REFERENCE
Koga,H. High-performance liquid chromatography measurement of antimicrobial concentrations in polymorphonuclear leukocytes, *Antimicrob.Agents Chemother.*, **1987**, *31*, 1904–1908.

SAMPLE
Matrix: solutions
Sample preparation: Prepare a 250 μg/mL solution of clindamycin in mobile phase, inject a 25 μL aliquot.

HPLC VARIABLES
Column: 250 × 4.6 Zorbax C8
Mobile phase: MeCN:water 12:88 containing 0.25 g/L tetrabutylammonium perchlorate and 2 mL/L 70% perchloric acid, apparent pH adjusted to 2.5 with 50% NaOH
Flow rate: 1.5
Injection volume: 25
Detector: UV 214

CHROMATOGRAM
Retention time: k' 0.9

OTHER SUBSTANCES
Simultaneous: benzyl alcohol, clindamycin, pirlimycin

REFERENCE
Theis,D.L. Ion-pairing liquid chromatographic method for the determination of pirlimycin hydrochloride, *J.Chromatogr.*, **1987**, *402*, 335–343.

SAMPLE
Matrix: tissue

Sample preparation: Condition a 3 mL 500 mg Sep-Pak Vac C18 SPE cartridge with 15 mL MeOH and 5 mL water. Homogenize 5 g blended fish tissue with 20 mL 10 mM pH 4.5 KH_2PO_4buffer at 8000 rpm for 2 min. Centrifuge the homogenized sample at 4000 g for 10 min. Decant and collect the supernatant, extract the residue with 20 mL buffer. Filter the combined extracts through glass wool. Add 1 mL 10% sodium tungstate solution and 1 mL 34 mM sulfuric acid, mix. Centrifuge at 4000 rpm for 15 min, filter the supernatant and discard the precipitated protein. Add 1mL 3% 1-pentanesulfonic acid and mix well. Add the fish extract to the SPE cartridge. Wash with 4 mL MeOH:water 10:90 and 2 mL water at 1 mL/min. Elute with 2 mL MeCN:water 50:50. Add 200 μL 1 M KOH and 500 mg NaCl to the eluate. Extract with three 3 mL portions of ethyl acetate by agitating on a Vortex mixer and centrifuging at 800 g for 3-5 min. Combine the extracts and filter through 3 g anhydrous sodium sulfate. Wash sodium sulfate with 2 mL ethyl acetate. Evaporate the filtrate to dryness under vacuum at 35°. Reconstitute the residue in 1 mL mobile phase, filter (0.45 μm). Inject a 50 μL aliquot.

HPLC VARIABLES

Column: 250 × 4.6 Spherisorb S5 ODS2
Mobile phase: MeCN:20 mM pH 4.5 potassium phosphate buffer containing 20 mM sodium 1-octanesulfonate 22:88
Flow rate: 1.0
Injection volume: 50
Detector: E, ESA Model 5100A, Model 5010 analytical cell, first electrode +0.65 V, second electrode +0.9 V, Model 5020 guard cell +0.95 V

CHROMATOGRAM

Retention time: 22
Limit of detection: 7 ng/g (muscle), 12 ng/g (skin)
Limit of quantitation: 17 ng/g (muscle), 24 ng/g (skin)

KEY WORDS

salmon; muscle; skin; SPE

REFERENCE

Luo,W.; Hansen,E.B.,Jr.; Ang,C.Y.W.; Thompson,H.C.,Jr. Determination of lincomycin residue in salmon tissues by ion-pair reversed-phase liquid chromatography with electrochemical detection, *J.AOAC Int.*, **1996**, *79*, 839–843.

SAMPLE

Matrix: tissue
Sample preparation: Condition a C18 Sep-Pak SPE cartridge with 20 mL MeOH and 40 mL distilled water. Homogenize 5 g chopped kidney with 50 mL MeOH, 5 g sodium sulfate, and 2 mL 1 M NaOH. Centrifuge at 1200 rcf for 10 min, decant the supernatant, repeat the extraction with another 50 mL portion of MeOH. Adjust the MeOH phases to pH 4 with 1 M HCl, filter (Whatman No.4 filter paper), transfer filtrate to a separatory funnel, add 50 mL hexane, shake. Remove the lower MeOH phase and reduce its volume to approximately 5 mL on a rotary evaporator. Transfer concentrated extract to a centrifuge tube with four 2 mL portions of water, add 50 mg sodium tungstate, centrifuge at 1200 rcf for 10 min. Add the supernatant to the SPE cartridge, wash with 4 mL water, elute with 5 mL MeOH, evaporate the eluate to dryness under nitrogen, redissolve the residue in 200 μL mobile phase inject an aliquot.

HPLC VARIABLES

Guard column: 50 × 5 30-35 μm Co Pell ODS
Column: 250 × 5 5 μm Spherisorb ODS
Mobile phase: MeOH:pH 7.0 phosphate buffer 65:35
Flow rate: 1
Injection volume: 200
Detector: UV 214

KEY WORDS

cow; pig; kidney; SPE

REFERENCE

Farrington,W.H.H.; Cass,S.D.; Patey,A.L.; Shearer,G. A method for the analysis of lincomycin in porcine and bovine kidneys, *Food Addit.Contam.*, **1987**, *5*, 67–76.